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3 MULTIDISCIPLINARY RESEARCH LEADING TO  
UTILIZATION OF EXTRATERRESTRIAL RESOURCES 4

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October 1, 1966 to January 1, 1967 9 1000

U. S. Bureau of Mines NASA Program of Multidisciplinary Research  
Leading to Utilization of Extraterrestrial Resources

QUARTERLY STATUS REPORT

October 1, 1966 to January 1, 1967

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## STATUS REPORT SECOND QUARTER FISCAL YEAR 1967

U. S. Bureau of Mines NASA Program of Multidisciplinary Research  
Leading to Utilization of Extraterrestrial Resources

January 1, 1967

Task title: Core group activity  
Investigator: Thomas C. Atchison, Senior Research Scientist  
Location: Twin Cities Mining Research Center  
Minneapolis-St. Paul, Minnesota  
Date begun: April 1965 To be completed: Continuing  
Personnel: Thomas C. Atchison, Supervisory Research Physicist  
David E. Fogelson, Research Geophysicist  
Clifford W. Schultz, Research Extractive Metallurgist  
Lowell W. Gibbs, Mining Methods Research Engineer  
Other Bureau personnel, as assigned

### PROGRESS REPORT

#### Objective

To provide the basic scientific and engineering knowledge needed for subsequent development of an extraterrestrial mineral resource extraction, processing and utilization technology for supporting and enhancing the economy of manned lunar and planetary missions.

#### Progress During the Second Quarter

The core group continued to obtain, evaluate, and distribute background information applicable to the program by literature search and direct contact with groups conducting related research.

At the request of NASA a meeting was arranged at the Twin Cities Center on November 30 to review the progress and future plans for the Bureau's extraterrestrial resource utilization program. A number of scientists and engineers from NASA headquarters and field laboratories attended and participated in discussion and evaluation of the work. The investigators in charge of each of the research tasks at seven different research centers involved in the program presented their work and took part in the discussion.

At the meeting members of the core group presented background information on the Bureau's program and explained its present structure. The presentation by each investigator included a description of the relation of his NASA task to his regular Bureau research to demonstrate the competence that is being brought to bear on the NASA problems. Following this was a brief highlight of the investigator's

progress or planned attack on his task. The meeting also included a general discussion period and a tour of the Twin Cities Center laboratories.

In December the core group submitted a proposal for continuing the Bureau's NASA program into its third and fourth years. The proposal describes the background, present status, and future plans for the program. It stresses the interrelation of the research tasks being conducted at a number of Bureau centers and the adaptability of the program to new information produced by the Bureau tasks or by related work of outside groups. Because of present budget restrictions on NASA, additional funds requested are limited to those needed to maintain the current funding of \$300,000 per year. However, the proposal points out that, when funds are available, other research tasks should be started to help meet the program objective.

#### Status of Manuscripts

Mining on the Moon, by C. W. Schultz, was published in NEW SCIENTIST, July 9, 1966, p. 33.

Materials Testing Laboratory for Operation on the Lunar Surface, an informal report by Thomas C. Atchison, was submitted to NASA in August.

Objective and Method of Attack for Bureau Extraterrestrial Resource Utilization Program, by Thomas C. Atchison, and Scheduling of Research Tasks for Bureau Extraterrestrial Resource Utilization Program, by Lowell W. Gibbs, were presented at the NASA review meeting at the Twin Cities November 30.

Proposal for Continuing Bureau Extraterrestrial Resource Utilization Program, by the core group, was submitted to NASA in December.

Bureau of Mines Research Using Simulated Lunar Materials, by Thomas C. Atchison and David E. Fogelson, a paper for presentation at the Northwest Metals and Minerals Conference, Portland, Oregon, April 19-21, is in preparation.

Task title: Selection and sample collection of simulated lunar materials  
Investigator: David E. Fogelson, Project Leader  
Location: Twin Cities Mining Research Center  
Minneapolis-St. Paul, Minnesota  
Date begun: September 1965 To be completed: Continuing  
Personnel: David E. Fogelson, Research Geophysicist  
Other Bureau personnel, as assigned

## PROGRESS REPORT

### Objective

Select and obtain samples of rocks and minerals covering the range of materials likely to be found on the Moon.

### Progress During the Second Quarter

Another rock type, a gabbro, was collected from northern Minnesota. The gabbro selected was from the well-known Duluth gabbro, one of the largest intrusions of gabbro in the world. This intrusive mass is believed to be a lopolith or a giant underground basin. Such structural features have led Paul Lowman to suggest the theory that the lunar maria may be similar to terrestrial lopoliths.

We have decided to add to our collection some shock metamorphosed material such as is found near impact craters or nuclear explosions. These may be either rocks fractured by shock or shock indurated sediments. Selection of representative materials presents a problem.

A talk summarizing the progress made on this project was presented at the NASA review meeting on November 30.

### Status of Manuscripts

Selection and Collection of Simulated Lunar Materials, by David E. Fogelson, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Physical properties of simulated lunar materials  
Investigator: Thomas C. Atchison, Senior Research Scientist  
Location: Twin Cities Mining Research Center  
Minneapolis-St. Paul, Minnesota  
Date begun: October 1965 To be completed: Continuing  
Personnel: All projects are participating

## PROGRESS REPORT

### Objective

To incorporate simulated lunar materials into basic fragmentation research currently in progress. By this means to determine the composition, elastic, strength, surface, thermal, electrical, magnetic, and explosive shock properties of simulated lunar materials in Earth environment.

### Progress During the Second Quarter

Specimens of granodiorite, gabbro, and two additional types of vesicular basalt were prepared and added to a number of the property measurements in progress. Measurements of the coefficient of rock strength and preliminary measurements of uniaxial compressive and tensile strengths on the original 10 simulated lunar materials were completed by the Mechanical Fragmentation group.

Evaluation of the effect of anisotropy on the strength and elastic properties of the rocks was continued by the Rock Physics group. Four-inch diameter spheres of pumice, rhyolite, obsidian, flood basalt, and vesicular basalt were pulsed, and equal area projections were made of calculated compressive wave velocities. The projections were contoured to illustrate the change in velocity with direction and to facilitate comparison with visible fabric orientation. Figures 1 and 2 show the results for the pumice. The maximum velocity contour of 3.50 km/sec coincides with the statistically preferred orientation of vesicle long axes in this rock. This technique of determining anisotropy by making sonic measurements on a sphere and projecting the results was presented at the NASA review meeting at the Twin Cities on November 30.

Work on measuring dielectric constants and dissipation factors by the Thermal Fragmentation group was rescheduled to be completed in the third quarter because the dielectric sample holder was returned late by the manufacturer after reconditioning and recalibration. The unit was received December 9, and the Q-meter and sample meter were checked using previously measured alumina specimens for comparison purposes. Measurements on 13 simulated lunar rocks are underway.

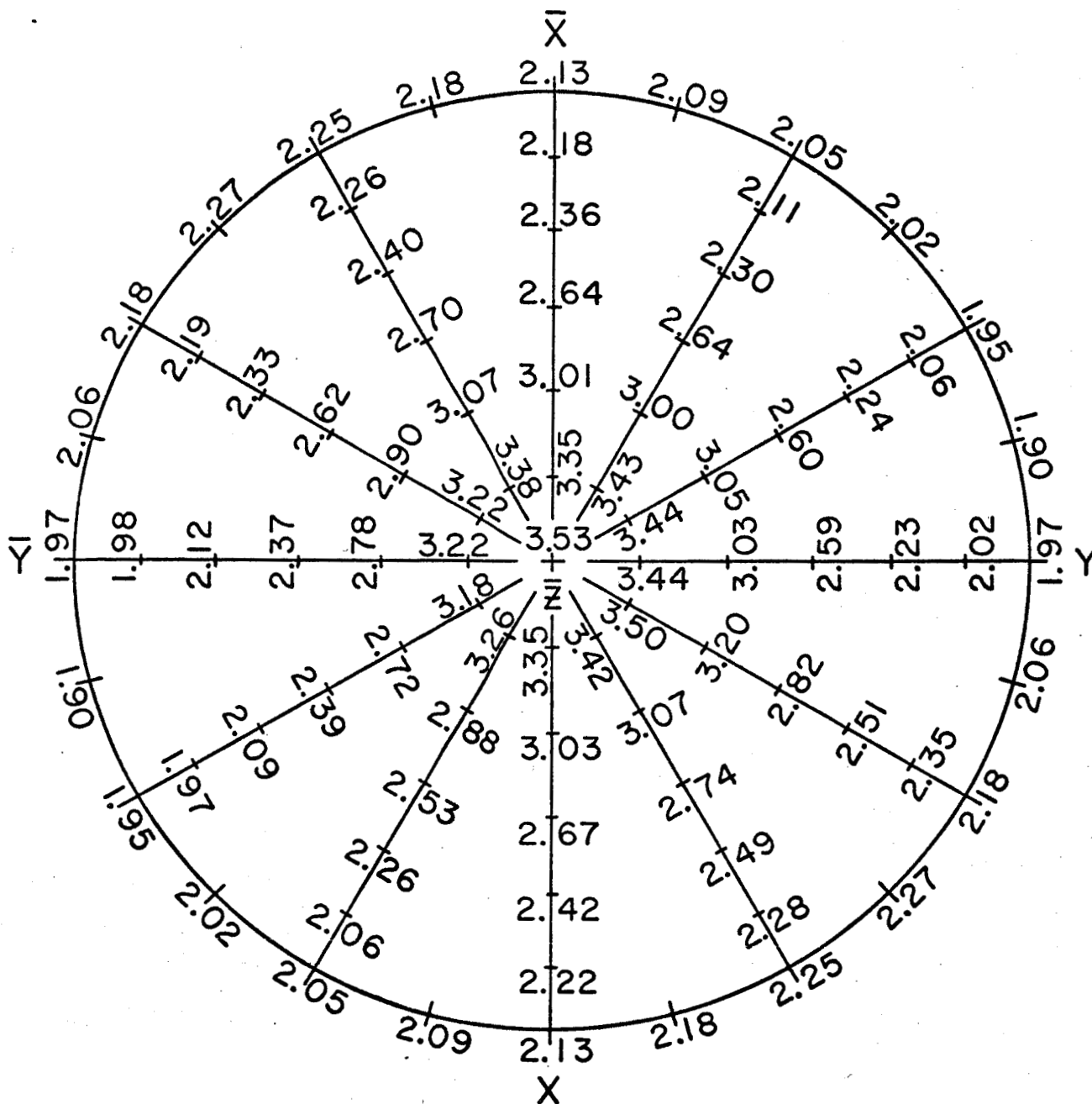


FIGURE 1. - Equal area projection of symmetrically-disposed compressional wave velocities in km/sec for Newberry crater pumice.

Lower hemisphere equal area projection

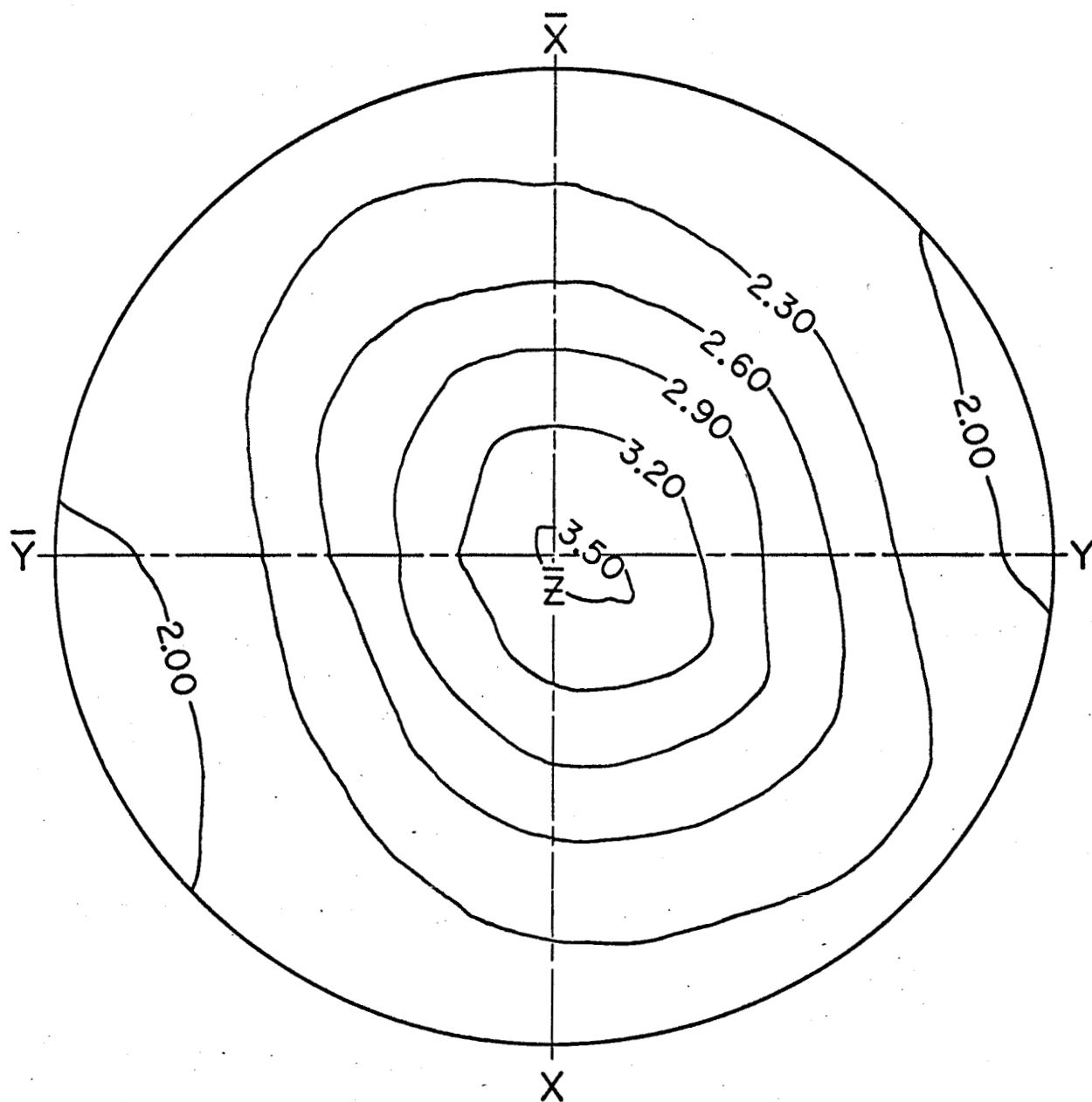


FIGURE 2. - Contoured compressional wave velocities  
in km/sec of Newberry crater pumice.

Lower hemisphere equal area projection



To show the range of property values represented by the simulated lunar materials we have selected, table 1 gives average values for some of the properties that have been measured for nine of the rocks. These data are preliminary and may be modified by additional work that takes into account the anisotropy that is characteristic of some of the rocks, as well as other refinements in measurement and analysis. The table illustrates the broad range of property values covered by the simulated lunar materials, extending beyond that usually encountered in mining problems on Earth at the low density, low strength, and low hardness end of the scale.

TABLE 1. - Properties of simulated lunar rocks in Earth environment

| Rock type        | Apparent density (g/cm) | Apparent porosity (percent) | Pulse velocity (km/sec) | Compressive strength (psi) | Tensile strength (psi) | Hardness (Shore units) |
|------------------|-------------------------|-----------------------------|-------------------------|----------------------------|------------------------|------------------------|
| Dunite           | 3.19                    | 1                           | 7,500                   | 27,000                     | 2,000                  | 73                     |
| Basalt           | 2.84                    | 2                           | 6,000                   | 53,000                     | 3,400                  | 84                     |
| Serpentine       | 2.56                    | 3                           | 6,000                   | 18,000                     | 880                    | 68                     |
| Obsidian         | 2.39                    | 1                           | 5,600                   | 65,000                     | 2,200                  | 103                    |
| Rhyolite         | 2.35                    | 8                           | 4,200                   | 22,000                     | 1,200                  | 79                     |
| Vesicular basalt | 2.25                    | 20                          | 3,800                   | 10,000                     | 1,100                  | 81                     |
| Dacite           | 1.98                    | 17                          | 4,500                   | 6,000                      | 620                    | 35                     |
| Tuff             | 1.15                    | 50                          | 2,500                   | 850                        | 100                    | 10                     |
| Pumice           | .76                     | 62                          | 2,500                   | 1,500                      | 240                    | 5                      |

Status of Manuscripts

None in progress.

Task title: (1) Chemical reactivity and cold welding of freshly formed surfaces  
(2) Surface properties of rock in lunar environment  
Investigator: Clifford W. Schultz, Project Leader  
Location: Twin Cities Mining Research Center  
Minneapolis-St. Paul, Minnesota  
Date begun: January 1966 To be completed: March 1969  
Personnel: Clifford W. Schultz, Research Extractive Metallurgist  
William H. Engelmann, Research Chemist  
Wallace W. Roepke, Physical Science Technician  
Sheldon L. Altman, Research Equipment Operator  
Ernest Bukofzer, Engineering Technician

## PROGRESS REPORT

### Objective

Measure the equilibrium constants for the adsorption of gases on the surfaces of silicate minerals. Relate this quantity to the fractional coverage necessary to inhibit cold welding and determine the rate at which various other processes inhibit or prohibit cold welding of vacuum formed surfaces. Extend current experimental studies of surface properties of rocks and minerals to include lunar environment.

### Progress During the Second Quarter

During the past quarter several outgassing tests have been made on one-inch cubes of semiwelded tuff and flood basalt. These tests are preliminary to an investigation of the gas loads and pumpdown characteristics of the simulated lunar rock samples. The purpose of this investigation is to determine the equilibration time for the samples in the simulated lunar environment and further to devise a preconditioning scheme which will minimize this time.

Typically, a preconditioning scheme might be as follows. It must take into consideration the lack of water vapor in the lunar environment. The lunar temperature of 130°C allows baking of the samples under vacuum for several hours as a drying procedure. After a suitable bakeout, as indicated by a decreasing pressure in the vacuum oven, the oven may be shut off and the sample allowed to cool. When the oven is to be opened it must be vented with room air through a drying column of Drierite or Linde sieve. Opening the oven will allow water vapor from the room to adsorb on the samples. However, since the interstitial voids are dry gas filled, it will be some period of time before this becomes absorption. With the test sample moved to the high vacuum chamber as rapidly as possible, there will be a minimum of water vapor contamination. The oven must also be closed promptly and pumped out again. No heating will be needed after the initial bakeout if the periods of time the oven is open are very short. A tray of Linde sieve in the bottom of the oven will help keep the chamber at its driest.

The results of the outgassing tests performed in the past quarter are summarized in figures 1 and 2. Figure 1 compares the pumping rate attained with an empty chamber (I) with that of the chamber containing a cube of semiwelded tuff (II). The difference in rates is taken to be the outgassing rate of the sample. Plotting the log of this difference, we find that it varies linearly with pressure (curve I, figure 2). The implication of this linear relationship is that desorption is the rate controlling mechanism in the outgassing process. Curve II which is the outgassing rate of the basalt sample is nonlinear and therefore has a different rate controlling mechanism, perhaps diffusion. From these and other observations the following inferences can be made:

1. The samples are somewhat hygroscopic, i.e., water is more strongly adsorbed than gases; this is demonstrated by higher pumpdown rates after pretreatment with dry gases. A more nearly quantitative answer will be obtained when the mass spectrometer is available for these studies.
2. The flood basalt may present a greater problem in the ultra-high vacuum system than the porous semiwelded tuff. This is evidenced by the higher outgassing rate at low pressures in figure 2.

The buildup of vacuum capability has proceeded with the receipt of the Ewald bench welder and the Precision Scientific Co. vacuum oven. The Ultek, Quad 250, mass spectrometer and the ultrasonic vapor degreaser from Alcar Instrument Co. have not yet been received.

The machining of all parts for the test fixture to be used in the friction studies is finished. The strain gage pickups must be assembled and a jig must be made for the bench testing. Cleaning and assembly for bench checking has already been started.

The Ultek ultrahigh vacuum system has passed acceptance tests. It is capable of attaining a pressure, when clean, dry, and empty, of better than  $5 \times 10^{-12}$  torr. The Ultek system now has the two rotary feed-throughs, required by the friction test fixture, in place. A portable cart is being fabricated to hold the steel bell jar when it is not in use. This cart will allow for low pressure to be maintained in the bell jar, thus keeping it dry and clean which will greatly reduce pumpdown times.

#### Status of Manuscripts

Chemical Reactivity and Cold Welding - Surface Properties, by C. W. Schultz, was presented at the NASA review meeting at the Twin Cities November 30.

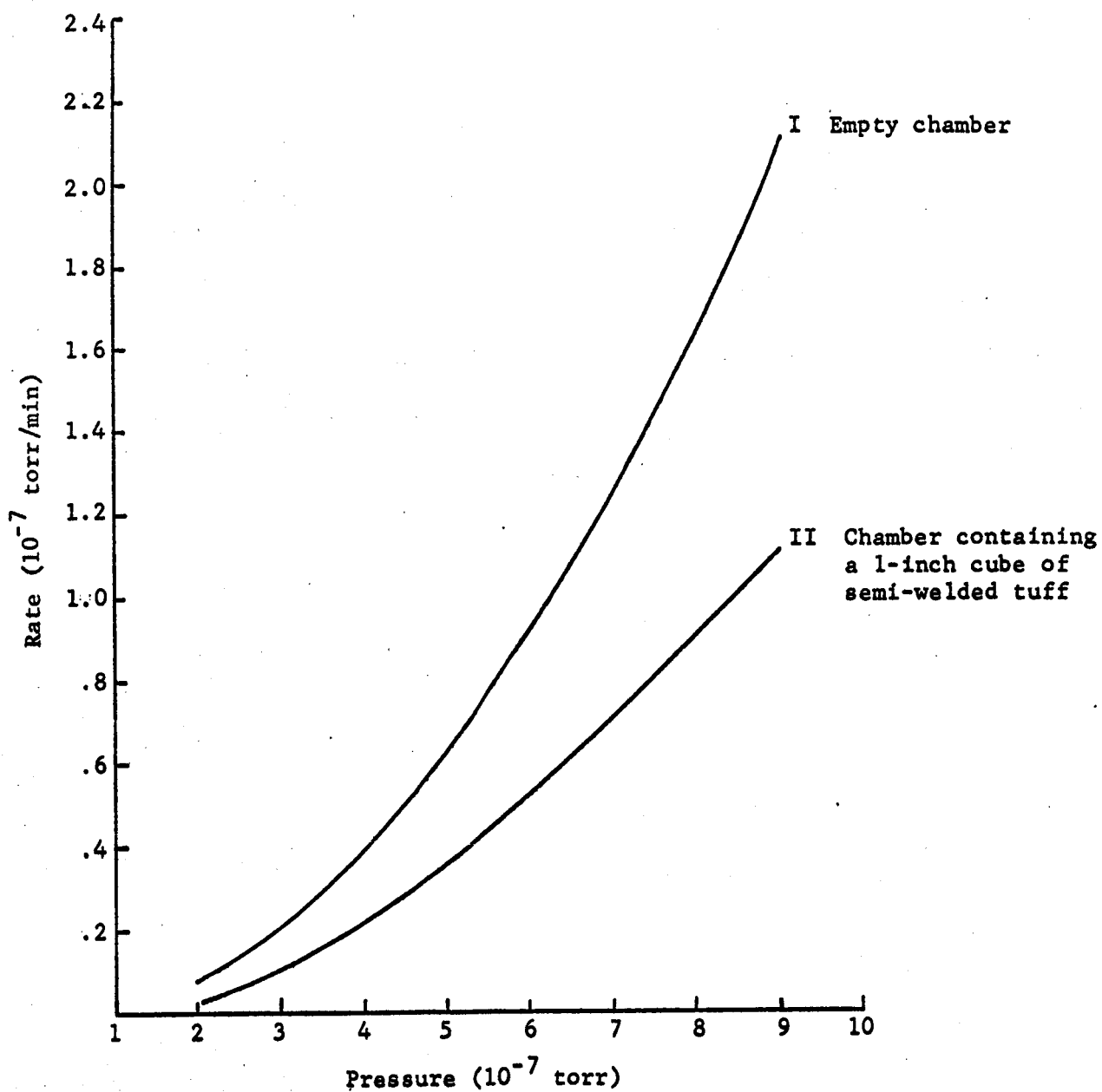


FIGURE 1. - A comparison of the pumping rates for an empty chamber and a chamber containing a sample of semi-welded tuff

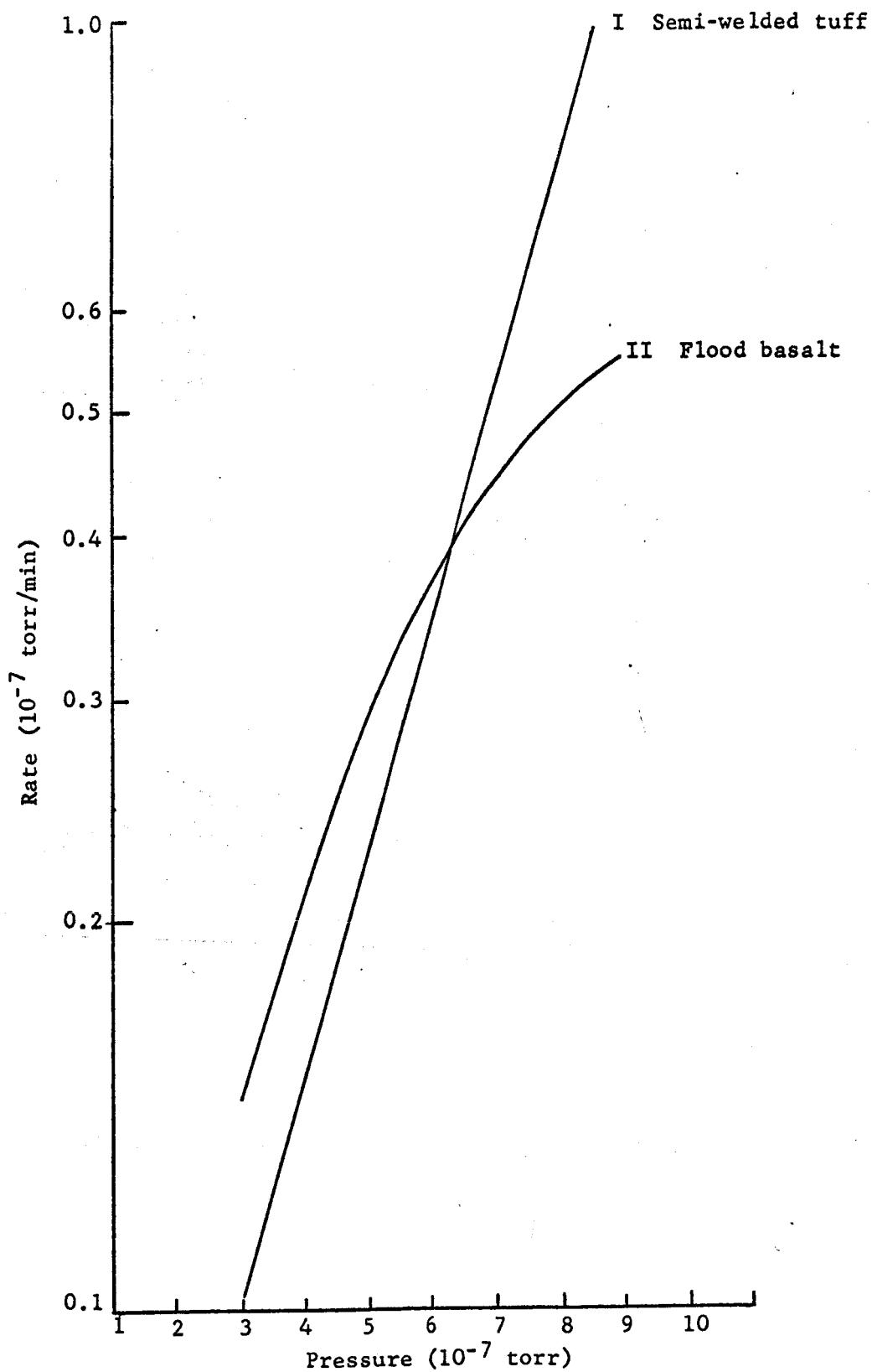


FIGURE 2. - Outgassing rates of semi-welded tuff and flood basalt as a function of pressure

Task title: (1) Fracture and other failure mechanisms in lunar environment  
(2) Strength and elastic properties of rock in lunar environment  
Investigator: John R. McWilliams, Project Leader  
Location: Twin Cities Mining Research Center  
Minneapolis-St. Paul, Minnesota  
Date begun: June 1966 To be completed: June 1969  
Personnel: John R. McWilliams, Mining Methods Research Engineer  
Robert J. Willard, Geologist  
Thomas R. Bur, Research Geophysicist  
Egons R. Podnieks, Mechanical Research Engineer  
Richard E. Thill, Geophysicist  
Peter G. Chamberlain, Geophysicist  
Kenneth E. Hjelmstad, Geologist  
Richard M. Brumley, Electronic Development Technician

## PROGRESS REPORT

### Objective

Extend current experimental studies of rock failure by such mechanisms as dislocation, twinning, and crack formation to include lunar environment. Extend current measurements of static and dynamic elastic moduli and compressive and tensile strengths of rock to include lunar environment.

### Progress During the Second Quarter

Progress on the study of the effect of environment on the static and dynamic properties of rocks was evidenced primarily in the area of upgrading equipment and measurement techniques. Actual measurements were not underway until near the end of the quarter.

The Ultek ultrahigh horizontal chamber vacuum system has been installed and checked out. It has been found to meet specifications in most respects, but final acceptance is awaiting adjustments in the bakeout heaters by the manufacturer. The system is undergoing adaption for use with sonic and static testing equipment. Specimen preconditioning requirements are being studied to insure that equilibrium within test specimens will be attained under test conditions of temperature, pressure, and humidity.

A digital-to-analog converter has proven reliable for making direct plots of stress versus velocity on an x-y recorder by means of a rapid start-and-stop pulsing system. This arrangement will provide nearly continuous monitoring of velocity changes in samples during static cycling tests, and will eliminate the visual recording of travel time and the manual plotting of an extremely large number of data points.

New transducers have been made for use with the above start-and-stop pulsing system. The transducers will allow greater flexibility in sample length and will exhibit greater stability under varying temperature conditions.

The environment control system for maintaining the three temperature levels and four kinds of atmosphere required for the current tests has been tested to establish its reliability and precision. It is now operating satisfactorily and the planned series of experiments has begun.

Of interest to future fracture studies under this task and under the following task related to thermal fragmentation is a preliminary surface and thin section study that we made of several rock bars that had been subjected to focused and unfocused laser beams by a research group at M.I.T. The beams caused surface and internal damage to the bars (granite and marble). The types of induced damage are (1) surface fusion of mineral constituents and (2) internal cracking within grains and/or along grain boundaries. The type and extent of damage depends upon the beam character. In general, the unfocused laser beam appears to have caused more internal damage than the focused beam. The type and extent of damage appears to be no different than damage induced on and in rock by other well-known thermal heat sources capable of concentrated energy in a small area as, for example, a blow torch.

#### Status of Manuscripts

Rock Failure Mechanisms - Strength and Elastic Properties, by J. R. McWilliams and R. J. Willard, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: (1) Rock vaporization, melting, and thermal fracturing methods in vacuum  
(2) Thermophysical, strength, and elastic properties of rock at elevated and reduced temperatures in vacuum  
Investigator: Robert L. Marovelli, Project Leader  
Location: Twin Cities Mining Research Center  
Minneapolis-St. Paul, Minnesota  
Date begun: October 1966 To be completed: September 1968  
Personnel: Robert L. Marovelli, Mining Methods Research Engineer  
Russell E. Griffin, Electronic Research Engineer  
Ta-Shen Chen, Mechanical Research Engineer  
Carl F. Wingquist, Physicist  
David P. Lindroth, Physicist  
Sam G. Demou, Physicist  
Daryl J. Jersak, Electronic Development Technician  
Walter G. Krawza, Electronic Development Technician

## PROGRESS REPORT

### Objective

Investigate the feasibility of extending current thermal fragmentation studies to lunar vacuum environment. Currently the thermophysical, strength, and elastic properties of rock at temperatures up to the melting point are being measured. Extend this work to the low temperature range of lunar environment. Investigate the feasibility of extending these property measurements to lunar vacuum environment.

### Progress During the Second Quarter

A study of the point-load breaking strength and the three-point bending strength of three rock types at room (75°F) and liquid nitrogen (-320°F) temperature was carried out at the University of Wisconsin. A factorial design experiment, using three variables - temperature, span, and diameter - at two levels, was made on a limestone rock to evaluate the effects of these variables on the mechanical properties of this rock. The same mechanical tests were applied to basalt and granite specimens supplied by this Center. In general, average increases in the point-load breaking strength at the low temperature level over room temperature level were 46 percent for the limestone, 52 percent for the basalt, and 21 percent for the granite. The average modulus of rupture strength increased 59 percent for the limestone, 49 percent for basalt, and 19 percent for granite. A determination of Young's modulus at the two temperature levels was made on the limestone. The increase in the modulus was approximately 2.3 times that at room temperature. A manuscript for outside presentation was completed on this work.



Diametral-compression tests were made on the simulated lunar rock suite of obsidian, pumice, tuff, vesicular basalt, dacite, rhyolite, dunite, flood basalt, granodiorite, and serpentinite. The percent increase in strength at -320°F compared to 75°F ranged from 17 percent for rhyolite to 87 percent for serpentinite. The significant strength increase of cold rock was reported by Marovelli at the November 30 NASA review meeting.

Qualitative thermal shock tests using the plasma torch on three-inch glass hemispheres and three-inch alumina hemispheres continued. A few comparative tests were also made on quartzite and obsidian to check for spalling and melting. The glass hemispheres were found to shatter on heating after a few seconds. If heat is applied for less time than it takes for shattering, a surface fracture pattern appears during air cooling with an underlying bubble formation. Damage comparison experiments were conducted with heating and water cooling cycles, and with delays between the two cycles.

In connection with the shattering tests, motion pictures of up to 8,000 frames/sec were obtained of alumina hemispheres fragmenting. In the case of the glass hemispheres, the film speed was 4,000 frames/sec. We experimented with different filters and films trying to obtain the best detail of the fracture pattern as it occurs. A stainless steel mirror was also used to view the opposite side of the hemisphere. Pictures taken at these speeds are not fast enough to show the propagation of the cracks, but much information can be obtained about the type of fracturing and the pattern it follows. Continuing efforts will be made to increase the motion picture speed capability and improve the detail obtainable.

As part of this effort, steps are being taken to synchronize the camera starting time with the event through the use of strain gages. These will be placed on the outer edge of the face of the hemisphere and should be useful from two different standpoints - triggering the camera by signaling a buildup of stress before fracture, and enabling analysis of the stress waves arising from the momentum of the jet, the incident heat flux, and cyclic heating effects. Thermal indicating points were placed on the face of two hemispheres exposed to the plasma jet at normal settings. These points indicated a temperature range near the edge of the face of 150°F to 300°F while the temperature on the curved surface was under 150°F. These temperature ranges should allow dynamic strain gages to function normally in the above areas.

Receipt of a portable 16-mm film processor with a capacity of 200 feet greatly expedites the use of high-speed motion pictures. The film can be processed immediately to determine if the desired event was satisfactorily pictured, eliminating a week or more of waiting for results when the film is sent out for developing.

The thermal shock furnace delivery has been delayed. Several progress reports from the manufacturer have been received indicating work on it is continuing and delivery, originally promised for August, can be expected soon.

#### Status of Manuscripts

Thermal Fragmentation - Properties at High and Low Temperatures, by R. L. Marovelli, was presented at the NASA review meeting at the Twin Cities November 30.

The Effect of Low Temperatures on Some Physical Properties of Rock, by R. W. Heins and T. O. Friz, was prepared for presentation at the University of Texas/AIME Drilling and Rock Mechanics Conference in Austin, Texas on January 26 and possible publication as a journal article.

Task title: (1) Cuttings removal in drilling in lunar environment  
(2) Cooling and lubricating bits in drilling in lunar environment  
Investigator: James Paone, Project Leader  
Location: Twin Cities Mining Research Center  
Minneapolis-St. Paul, Minnesota  
To begin: January 1967 To be completed: December 1969  
Personnel: James Paone, Mining Methods Research Engineer  
Dick L. Madson, Mining Methods Research Engineer  
Robert L. Schmidt, Mining Engineer  
Vacancy, Physicist  
Kenneth G. Pung, Electronic Development Technician  
David. A. Larson, Engineering Technician

## PROGRESS REPORT

### Objective

Investigate various means of removing drill cuttings with and without flushing media in lunar environment. Investigate problems of heat removal and bit lubrication associated with drilling in lunar environment.

### Progress During the Second Quarter

In anticipation of the study of drilling in a lunar environment scheduled to begin next quarter, work plans were formulated and required equipment ordered. A technician was trained on instrumenting vacuum chamber experiments.

During the quarter, assistance was requested and provided to NASA for the Apollo Lunar Surface Drill program. The project leader attended a two-day meeting held in Baltimore, Md. as a member of the advisory commission on the technical evaluation of the Apollo Lunar Surface Drill program.

Work plans for the rest of the fiscal year follow.

## SCHEDULE OF WORK

### Third Quarter

Continue measurement of physical properties of simulated lunar rocks already underway. Compile a list of problem areas associated with the bit performance of the 30-meter and the 3-meter lunar drill systems, particularly those on cuttings, removal and bit cooling; evaluate previous work by NASA contractors and others on chip removal and bit characteristics.

Start drilling experiments in Earth atmosphere to ascertain temperatures generated in rotary, percussive, and rotary-percussive bits. The experiments will be performed in several different rocks to determine what effect the rock type may have on the temperature generated in the bits.

Concurrently, study different cuttings removal systems and incorporate one or more of the systems in the drilling experiments.

Start study of vacuum techniques particularly as they may pertain to drilling experiments in existing vacuum chamber facilities.

#### Fourth Quarter

Continue drilling and chip removal experiments in Earth atmosphere; begin modification of drill apparatus and instrumentation for drilling experiments in a simulated lunar environment.

#### MAJOR EQUIPMENT REQUIREMENTS

Instrumented drills (available), 4-channel recorder (available), bits (to be designed and purchased), ultrahigh vacuum system (may be purchased).

#### Status of Manuscripts

Cuttings Removal and Bit Cooling in Lunar Drilling, by James Paone, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Effect of lunar environment on behavior of fine particles  
Investigator: David E. Nicholson, Project Leader  
Location: Spokane Mining Research Laboratory  
Spokane, Washington  
Date begun: April 1966 To be completed: March 1969  
Personnel: David E. Nicholson, Mining Methods Research Engineer  
William R. Wayment, Mining Methods Research Engineer  
Dennis J. Kelsh, Physical Chemist  
Robert G. Parker, Chemist

## PROGRESS REPORT

### Objective

Extend current studies of fine particle behavior in mine backfill applications to include lunar environment. Measure such properties as density of packing, repose or friction angles, and rates of flow through orifices or channels. This work will be correlated with the study of electrostatic properties of granular particles being conducted at College Park, Maryland.

### Progress During the Second Quarter

Property measurements on fine particles classified into various size bands were halted during the quarter because of the transfer of project equipment to new laboratory space and because of other work demands on the project leader. The equipment is now being installed in the new space and the project leader expects to be able to spend full time on the fine particle studies soon after the first of the year.

Some background study on work related to lunar soil problems conducted by other groups was continued. Our future vacuum equipment needs were reviewed with personnel at the Twin Cities Mining Research Center. Any decision on the purchase of vacuum equipment will be delayed until after project personnel attend a vacuum technology short course at the University of California in Los Angeles in February. By that time we will have progressed further in designing our property and behavior tests, in selecting and preparing our test materials, and in conducting tests under a number of environments more easily obtained than lunar vacuum. By limiting and simplifying the tests that we need to make in lunar vacuum we may be able to have them performed in one of the vacuum systems at the Twin Cities Center. Another alternative to the immediate purchase of our own equipment would be the use of other government or industrial facilities on a cooperative or rental basis.

### Status of Manuscripts

Behavior of Fine Particles, by David E. Nicholson, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Support for underground lunar shelter  
Investigator: Ernest L. Corp, Project Leader  
Location: Spokane Mining Research Laboratory  
Spokane, Washington  
Date begun: April 1966 To be completed: March 1969  
Personnel: Ernest L. Corp, Mining Methods Research Engineer  
Robert C. Bates, Mining Methods Research Engineer

## PROGRESS REPORT

### Objective

The ultimate objective of this project is to advance the ground support technology needed to carry on extraterrestrial mining in support of space missions. The immediate objectives are: (1) To define the problems which will be encountered in designing a lunar ground support or environmental shelter system; (2) to investigate possible materials (indigenous and transported) which can fulfill the requirements for utilization in a support or environmental shelter system; (3) to formulate design concepts for support systems utilizing the most favorable materials.

### Progress During the Second Quarter

An attempt was made to subdivide the different types of underground lunar shelters into various categories depending on the controlling factors in their design. Since these factors are dependent on the depth of burial, the structures were categorized according to depth.

The first category includes structures built on the lunar surface or down to a depth of 25 feet. In this region design would be controlled almost completely by the requirements for adequate radiation and meteorite protection. Below 25 feet of depth the protection provided by the mass of lunar overburden would be equivalent to that provided by an Earth's atmosphere.

The second category includes structures built between 25- and 150-foot depth. In this region an internal pressure within the structure of one atmosphere would offset any of the pressures resulting from overburden. A structure or opening in this region would require only a seal to retain the one atmosphere of pressure.

Structures in the third category, which includes everything below 150 feet, must be designed to withstand overburden loads in addition to being sealed.

For the majority of lunar shelter construction (except those in excess of 150-foot depth), the problem can be reduced to one of optimum materials selection rather than optimum structural design. For this reason, the investigation and compilation of data on possible ground support materials (transported and indigenous) will be given considerable emphasis during the remainder of the fiscal year.

Another important consideration to be taken into account in selecting materials for a lunar shelter is the conditions under which construction will take place (vacuum or pressure). This will determine to a large degree the type of supporting material which could be used and the manner in which it is placed. One of the important factors controlling these construction conditions is the gas permeability of the surrounding lunar rock or soil. It must be assumed for the present that any possible value of permeability could exist. Therefore, the investigation of possible construction materials must include those adaptable to placement in a vacuum as well as at positive pressure.

The solution to the problem of designing structures for depths in excess of 150 feet in a lunar soil can to some extent be aided by the research work on the use of distributed-load support presently in progress at this laboratory. If a successful prediction equation for determining the loads acting directly on underground continuous structures (with known stress field) can be developed from planned model studies on buried flexible liners, this equation could conceivably be modified to determine the expected loading on underground lunar structures. This could be done by determining the physical constants of various simulated lunar soils.

The limited time devoted to this work to date has indicated that an extensive amount of background work still remains to be done before any specific test work is started. For this reason any decision on the purchase of a vacuum chamber will be postponed to later in the fiscal year or possibly next fiscal year. Therefore, the actual environmental test work will be deleted from the year's work plans.

#### Status of Manuscripts

Support for Underground Lunar Shelter, by Ernest L. Corp, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: (1) Effect of vacuum on explosive properties  
(2) Effect of micrometeoroid bombardment on explosives  
(3) Explosive blast effects in lunar environment  
Investigator: Frank C. Gibson, Project Coordinator, Explosive Physics  
Location: Explosives Research Center  
Pittsburgh, Pennsylvania  
Date begun: July 1966 To be completed: June 1969  
Personnel: Frank C. Gibson, Supervisory Research Physicist  
J. Edmund Hay, Research Physicist  
Richard W. Watson, Research Physicist  
Samuel R. Harris, Research Chemist  
Charles R. Summers, Research Physicist  
William F. Donaldson, Research Physicist  
Elva M. Guastini, Explosives Equipment Operator

## PROGRESS REPORT

### Objective

To develop a body of knowledge relevant to the use of chemical high explosives under lunar environment. Immediate goals are to determine the hazards associated with the storage, handling, and use of explosives in an environment characterized by high vacuum, extreme temperature cycling and a flux of small hypervelocity particles, and to establish techniques for minimizing these hazards.

### Progress During the Second Quarter

The immediate objective is the assembly of a facility in which the changes induced by temperature excursions in high vacuum can be studied and a separate facility comprising an adequate high-vacuum chamber capable of withstanding the detonation of relatively large explosive charges from which the sensitivity and performance of the explosive and initiator can be determined. Some preliminary experiments have been conducted relating to changes in the sensitivity of a typical explosive (Composition B) when exposed to moderate vacuum ( $10^{-3}$  torr); to date, no appreciable effect was observed. The overall problem has been clarified meanwhile by the standardization of a particular design of detonator and a particular explosive, hexanitrostilbene (HNS), for Apollo missions.

While the completion of the projected high-vacuum facility and the delivery of a quantity of the candidate explosive and initiator assembly are pending, the experimental effort has been directed to a study of the effect of high vacuum on the detonation characteristics of a widely used military explosive, Composition B. One of the most important parameters of an explosive, its critical diameter (the minimum diameter of a cylindrical charge in which detonation can propagate), is related to the sensitivity of the explosive and if the latter



is affected by environmental changes, the effect should appear in the critical diameter. Approach to the critical diameter is usually associated with a drop in the detonation velocity. To this end, cylindrical charges of Composition B, 1.9 cm in diameter and 10 cm long, were detonated in a vacuum of ca  $10^{-3}$  torr and the detonation velocity (determined by an electronic technique which gives a continuous record) compared with that at atmospheric pressure; no significant difference was found. Experiments to directly determine the critical diameter, using conical charges of cast Composition B (2.1 cm base diameter and 10.7 cm long) and loose granular tetryl (2.9 cm base diameter and 12.7 cm long), and attempting to locate the distance along the charge at which detonation ceases, have failed to show a clear cutoff point at either atmospheric or reduced pressure, indicating the need for improved resolution of the experimental technique.

An initiator system and prime candidate base charge have been chosen by NASA for the Apollo program. One of three candidate detonators has been qualified by NASA, Houston; it consists of a hermetically sealed initiator section that is mated with a base charge section having an O-ring seal; a lead azide primer and an HNS-I base charge are employed. The initiator is a hermetically sealed device specified to have a leak rate not to exceed  $10^{-6}$  cc per second at one atmosphere pressure differential. The explosive chosen for the base charge, 90 percent hexanitrostilbene (HNS) and 10 percent Teflon, is capable of withstanding long-term temperature excursions from  $-75^{\circ}\text{C}$  to  $+150^{\circ}\text{C}$  with only slight change in sensitivity.

Our effort in this project, in addition to determining the behavior of the expanding detonation products cloud (blast wave), is directed toward determining whether this initiator and explosive show evidence of impaired performance due to physical or chemical changes that would result from the high vacuum and temperature excursions of extraterrestrial environment. Since such changes may have a long-term effect which cannot be appreciated in the relatively short exposure time to which experiments must be limited, an attempt will be made to determine the nature and degree of any effect by X-ray crystallography and thin-film chromatography. The effectiveness of methods for preventing degradation will also be investigated.

#### Status of Manuscripts

Use of Explosives on the Moon, by Frank C. Gibson and J. Edmund Hay, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Volcanism and ore genesis as related to lunar mining  
Investigator: Rolland L. Blake, Project Coordinator  
Location: Twin Cities Metallurgy Research Center  
Minneapolis-St. Paul, Minnesota  
Date begun: June 1966 To be completed: May 1967  
Personnel: Rolland L. Blake, Research Geologist  
Others as assigned

## PROGRESS REPORT

### Objective

Study the genesis of ore deposits and the occurrence of minerals associated with volcanic activity here on Earth. Study the effects of the lunar environment and other environments on mineralization and ore genesis. Bring together the pertinent information found in the literature on these subjects and define those specific areas where additional work is needed.

### Progress During the Second Quarter

During the past several months three field trips were made and as a result the literature search proceeded at a reduced rate. The literature search has brought attention to the large number of uncommon terms that are used to describe volcanic features, processes, and products and the possible need for a glossary of volcanism terms. At present many of these terms are found in one of the several glossaries, dictionaries, and compilations of terms covering the broad field of geology. However, the increasing awareness of the importance of volcanism on the lunar surface, and the large number of scientists in space programs who are without formal training in volcanology, are factors supporting the need for a modern glossary that covers only the discipline of volcanism.

Cuyuna District Field Trip - On August 31 and September 1, accompanied by visiting Professor S. O. Agrell of the University of Cambridge, England, the investigator visited the open pit mines of the Cuyuna district in east-central Minnesota to examine the iron formation and the overlying partly volcanic rocks. Discussion on ancient volcanism associated with the Cuyuna rocks centered around the source of large quantities of iron furnished to the Precambrian basin of deposition. Professor Agrell recently conducted a seminar at the University of Minnesota on the Tertiary Volcanic Province of Great Britain.

California-Oregon Field Trip - The investigator accompanied Messrs. Atchison, Fogelson, and Schultz of the Twin Cities Mining Research Center on the second field trip from September 16 through October 4. Purpose of this trip was to discuss selection, collection and property testing of simulated lunar rock materials with scientists similarly

engaged, and to collect a number of rock samples for the Bureau's NASA program on extraterrestrial resources. The objectives for the task on volcanism were to discuss problems, work plans, and progress with volcanologists, and to visit extinct volcanic areas to examine rocks and collect samples for petrographic studies. Among those scientists visited in the Los Angeles area was Dr. Jack Green, Douglas Advanced Research Laboratories, who had previously offered his assistance. Dr. Green has extensively studied volcanic calderas, large depressions formed by collapse of surface volcanic rocks undermined by the outpouring of lava in large volumes. We discussed the possibility of his providing a report summarizing the mineral associations and ore deposits found in calderas.

Other visits with discussions were made in the San Francisco area at the USGS Branch of Astrogeology, the Stanford Research Institute, the University of California at Berkeley, and the California Division of Mines and Geology. Following these discussions, Atchison, Fogelson, and Blake visited a number of field sites in northern California and in southern and central Oregon. Excellent examples of extinct volcanic mountains, lava flows, cinder cones, pumice-, ash-, and tuff-beds, lava cones, and active solfatara springs were studied. The experience gained from closely examining the rocks, collecting samples and discussing their origin in the field has greatly increased our comprehension of the volcanic processes and their products. About five tons of rocks were collected for materials testing.

At the new Volcanology Center, University of Oregon at Eugene, discussions were held with the Director, Dr. McBirney and his visiting colleague, retired Professor Howel Williams of the Berkeley campus. Professor Williams was approached about contributing from his long experience as a volcanologist to the volcanism task. He did mention a European publication which he suggests may have an adequate glossary on volcanology terms. This reference has been ordered for the station library.

Hawaiian Islands Field Trip - The investigator made the third field trip to study volcanic processes and rocks in the active volcanic area along the Hawaiian rift zones. The trip was scheduled during the period November 14-27 to enable the investigator to attend the technical sessions of the GSA (Geological Society of America) Annual Meeting in San Francisco, and also to attend the GSA-sponsored post-meeting tour of three Hawaiian Islands. This tour was guided by leading volcanologists and all travel was arranged by a travel agency at a substantial cost reduction. Attendance at the technical sessions November 14-16 emphasized papers presented on volcanology and lunar surface materials. The Hawaiian Islands tour occupied the four days of November 17-20, in which volcanic cones, shield volcanoes, calderas, numerous lava flows, fire pits and pit craters on the islands of Oahu, Hawaii, and Maui were studied.

Status of Manuscripts

Volcanism and Ore Genesis, by Rolland L. Blake, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: (1) Reduction of silicates with carbon  
(2) Reduction of silicates in plasma torch  
Investigator: Sanaa E. Khalafalla, Acting Project Coordinator  
Location: Twin Cities Metallurgy Research Center  
Minneapolis-St. Paul, Minnesota  
Date begun: June 1966 To be completed: May 1969  
Personnel: Sanaa E. Khalafalla, Supervisory Research Chemist  
Larry A. Haas, Research Chemist

## PROGRESS REPORT

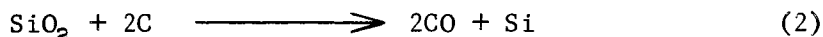
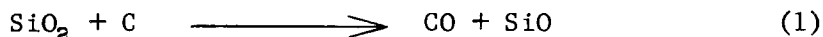
### Objective

To determine the optimum operating conditions for the extraction of oxygen from possible lunar raw materials in a high-temperature vacuum system. The research will establish the reduction rate of pure silica by graphite as a function of temperature, pressure, reactants ratio, and particle size. In addition, the reaction rate will be determined for other silica-bearing minerals using various carbonaceous reductants. The relative activity of the raw materials will be useful to ascertain the reactant characteristics which are most amenable for maximizing the reaction rate.

The immediate goal is to determine the effect of temperature on the graphite-silica reaction. Continued studies will devise an experimental procedure for determining the reaction rate at high reaction velocities without sample fluidization or blowout during the test. Also, the feasibility of reducing silicates with activated hydrogen in a plasma torch will be studied.

### Progress During the Second Quarter

Research has been directed to obtain a baseline which can be used as a reference in comparing the effects of various physicochemical variables on the high-temperature vacuum reduction reaction. Two possible reactions for carbonaceous reduction of silica have been suggested in the literature.<sup>1</sup>



For the experiments in this report, equimolar quantities of graphite and silica were used. The charge was prepared by thoroughly mixing

<sup>1</sup>Beecher, N., and R. E. Rosensweig. Ablation Mechanisms in Plastics with Inorganic Reinforcement. ARS Journal, v. 31, 1961, p. 537.

12 grams of minus 200- plus 270-mesh silica and 2.4 grams of minus 400-mesh ultrapure graphite. The evacuation, heating and cooling procedure was previously described in the first quarter report of fiscal year 1967. Samples were weighed and analyzed for their initial and final silica and carbon contents.

The test conditions and experimental results are presented in table 1. The reduction rate (percent of total oxygen removed) was calculated from the amount of reacted carbon, assuming all the carbon losses were consumed to form carbon monoxide. The calculated weight loss was difficult to determine since some of the reduced silica product apparently volatilized. The presence of Si and/or SiO in the residue has been verified but a quantitative analytical technique for determining metallic Si or SiO has not been devised. The calculated weight loss was determined, assuming all the reaction products were volatilized. Fair agreement between calculated and experimental weight loss was obtained, assuming SiO formation according to reaction 1. Evidence of SiO formation and volatilization was substantiated by chemical analysis of the condensate on the 800°C molybdenum radiation shields.

The dependence of reaction rate on temperature is shown in figure 1, where percent oxygen removed is plotted against temperature. All the tests were performed in the vacuum range of  $10^{-2}$  to  $10^{-4}$  torr.

The nonreproducibility of the results for experiments above 1,300°C was primarily due to sample blowout. Verification of this action was evident from the unreacted sample observed on the radiation shields and crucible pedestal. Efforts were continued to develop a technique to obtain replicable experimental results with minimum sample blowout. Apparently large amounts of gaseous products ejected an appreciable portion of unreacted carbon and silica from the crucible. This was especially noticeable at high test temperatures (above 1,300°C). It was not possible to place a gas-tight cover over the sample container since the pressure directly above the sample must be very low for a perceptible reaction rate. Attempts were made to design and construct a baffle-type cover with an indirect exit and minimum gas flow resistance. However, with the use of the baffle system, sample fluidization by CO evolution was still evident above 1,300°C using loose sample powders. Blumenthal<sup>2</sup> reduced the sample blowout problem by briquetting the charge mixture. A second series of experiments were performed using briquetted samples. The briquets were formed at room temperature under 50 tons per square inch pressure. The pressed samples were cylinders of 0.8 inch in diameter and height. Some evidence of sample blowout still persisted using the briquets.

<sup>2</sup>Blumenthal, J. L., M. J. Santy, and E. A. Burns. Kinetic Studies of High Temperature Carbon-Silica Reactions in Charred Silica-Reinforced Phenolic Resins. Paper presented at Western States Section of the Combustion Institute, Oct. 25-26, 1965, p. 12.

TABLE 1. - Temperature effect for carbon-silica reaction

| Test No. | Sample           | Reaction time, hr | Temperature ° C | Pressure range torr X 10 <sup>-3</sup> | Chemical analysis  |      | Oxygen removal rate <sup>1/</sup> percent/min | Weight loss, percent/hr  |          |
|----------|------------------|-------------------|-----------------|--|--------------------|------|---|--------------------------|----------|
|          |                  |                   |                 |  | % SiO <sub>2</sub> | % C  |   | Calculated <sup>2/</sup> | Observed |
|          | Charge mix       | --                | ----            | -----                                  | 83.3               | 16.7 | 0.000   | ----                     | ----     |
| 19       | Loose Powder     | 22                | 1110            | 2-0.2                                  | ----               | ---- | negligible                                    | negligible               | 0.00     |
| 11       |                  | 5                 | 1170            | 1-0.1                                  | 83.3               | 16.7 | do  | do                       | 0.02     |
| 14       |                  | 5                 | 1225            | 1.5-0.9                                | 83.2               | 16.4 | 0.001   | 0.20                     | 0.12     |
| 22       |                  | 22                | 1240            | 5-1.4                                  | 82.8               | 16.1 | 0.004   | 0.21                     | 0.11     |
| 21       |                  | 24                | 1280            | 5-9                                    | 87.5               | 13.7 | 0.016   | 1.07                     | 1.04     |
| 15       |                  | 5                 | 1280            | 3-4                                    | 83.6               | 16.4 | 0.022   | 1.04                     | 0.81     |
| 16       |                  | 5                 | 1300            | 1-15                                   | 85.5               | 15.9 | 0.075   | 4.62                     | 2.70     |
| 20       |                  | 22                | 1300            | 6-18                                   | 94.8               | 12.2 | 0.045   | 2.23                     | 2.05     |
| 26       |                  | 5                 | 1340            | 4-12                                   | 91.2               | 13.8 | 0.120   | 4.76                     | 5.51     |
| 21       | Briquets         | 24                | 1280            | 5-9                                    | 88.2               | 10.0 | 0.018   | 1.07                     | 1.04     |
| 24       |                  | 5                 | 1290            | 3-1                                    | 82.8               | 16.2 | 0.020   | 0.89                     | 0.41     |
| 20       |                  | 22                | 1300            | 6-18                                   | 94.8               | 12.2 | 0.045   | 2.23                     | 2.05     |
| 25       |                  | 5                 | 1340            | 4-20                                   | 94.7               | 12.0 | 0.167   | 7.10                     | 5.48     |
| 27       |                  | 5                 | 1345            | 10-30                                  | 97.7               | 10.9 | 0.200   | 8.05                     | 7.40     |
| 29       | Dextrose Pellets | 5                 | 1340            | 8-22                                   | 89.5               | 13.3 | 0.167   | 7.86                     | 6.92     |
| 30       |                  | 5                 | 1340            | 15-32                                  | 97.4               | 11.4 | 0.222   | 10.30                    | 10.00    |

<sup>1/</sup> Percent oxygen based on the total oxygen content in the charge.

<sup>2/</sup> Calculated on the basis of the change in carbon content, assuming all the reaction products were volatilized (CO and Si or SiO).

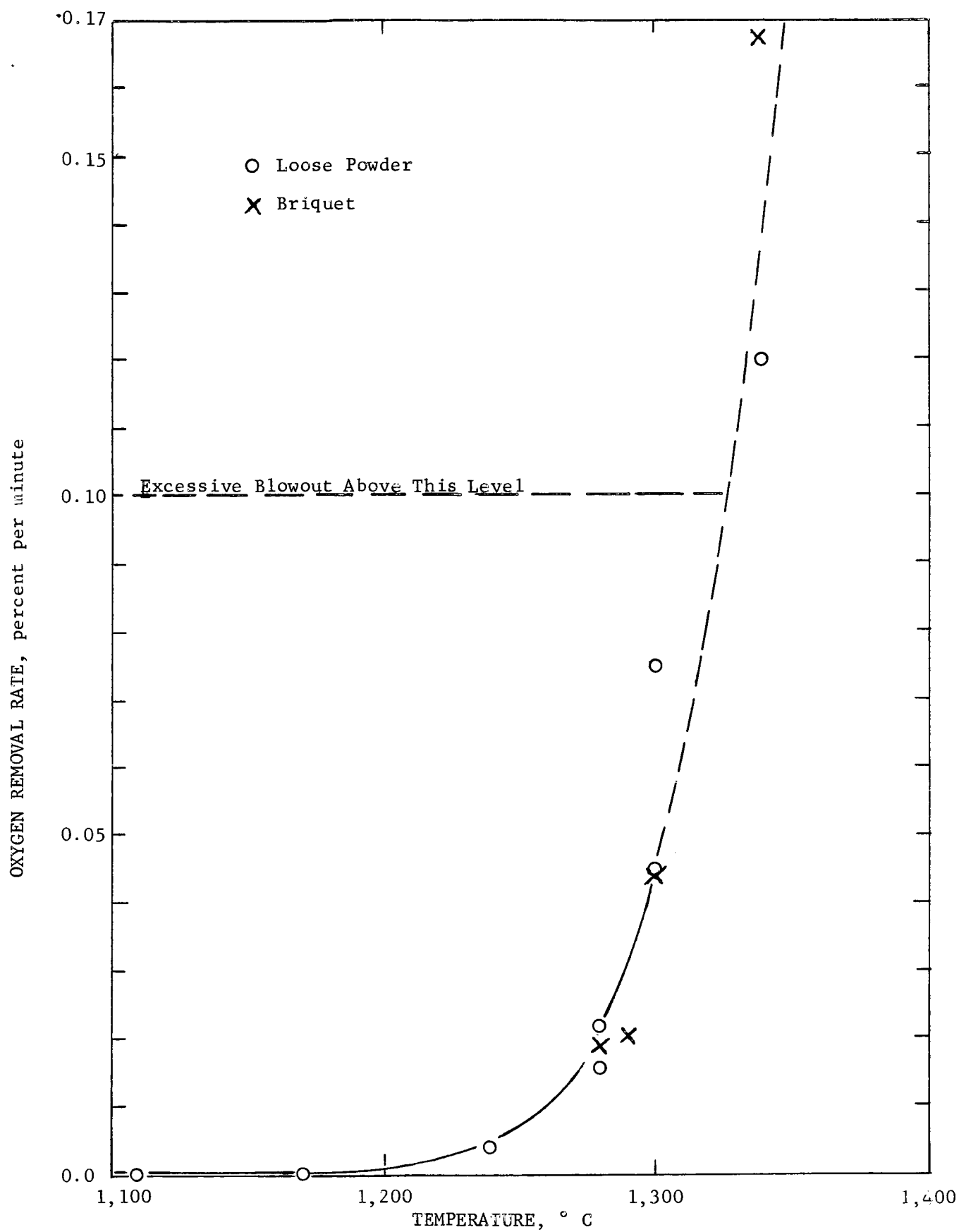


FIGURE 1. - Temperature effect on the oxygen removal rate.



A third series of experiments were performed adding to powder sample 1 to 2 percent dextrose as a pellet binder. Five to 10 cc of water was used to agglomerate the pellets. The dextrose was then decomposed to carbon at 500°F in air. This technique also did not produce satisfactory sample retention during the high temperature reaction in the vacuum furnace. Experimental difficulties were also encountered during sample preparation and the sugar carbonization roast.

A fourth series of experiments were performed using samples containing larger particles, 12 grams of minus 80- plus 120-mesh silica and 2.4 grams of minus 100- plus 200-mesh graphite. Higher test temperatures could be reached before blowout was observed. Experiments are in progress to determine the reaction temperature limit using larger particles.

To date, test results have indicated that the maximum reaction rate (with satisfactory sample retention) is approximately 0.1 percent reduction per minute. This corresponds to a carbon monoxide evolution flow rate of about 600 liters per minute at 1,423°C and 0.038 torr. With a flow of this magnitude, a low resistance filter system should be able to contain the powdered sample in the crucible. It is, therefore, anticipated that a quartz wool filter will solve the sample blowout problem.

Dr. Weston, the original investigator for these tasks, has left the Bureau for a position with private industry. Dr. Khalafalla has taken over supervision of the work. Investigation of the feasibility of mineral reduction in a high temperature plasma arc, originally planned for this quarter, has been rescheduled for completion in the third or fourth quarter.

#### Status of Manuscripts \*

Reduction of Silicates with Carbon, by Larry A. Haas, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Magnetic and electrostatic properties of minerals in  
a vacuum  
Investigator: Foster Fraas, Project Leader  
Location: College Park Metallurgy Research Center  
College Park, Maryland  
Date begun: June 1966 To be completed: May 1969  
Personnel: Ray A. Heindl, Supervisory Chemical Research Engineer  
Foster Fraas, Research Metallurgist

#### PROGRESS REPORT

##### Objective

Study adsorption and contact electrification in a vacuum and determine their effect on the separability of nonconducting minerals.

##### Progress During the Second Quarter

Work during the second quarter involved the design of vibrating electrifiers with vibration transmitted through bellows seals. After considerable delay the vacuum pump was delivered. Progress has been slowed by the delay in delivery of equipment. However, the schedule outlined in the original work plan is expected to be maintained, with the next quarter including assembly of equipment, and measurement of the relative extent of interfering and useful electrification effects.

##### Status of Manuscripts

Electrostatic Properties in Vacuum, by Foster Fraas, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Biological production of sulfuric acid  
Investigator: Joseph A. Sutton, Project Leader  
Location: College Park Metallurgy Research Center  
College Park, Maryland  
Date begun: June 1966 To be completed: May 1967  
Personnel: Ray A. Heindl, Supervisory Chemical Research Engineer  
Joseph A. Sutton, Research Chemist  
John D. Corrick, Research Chemist  
Jerry M. Carosella, Microbiologist

## PROGRESS REPORT

### Objective

Establish the limiting environmental conditions for the survival of bacteria of the genus thiobacillus. Determine the rate of sulfuric acid production within these limits. Conduct a literature survey and visit such laboratories as may be necessary to establish the state of the art in the use of bacteria in any stage of a life support system in an extraterrestrial environment.

### Progress During the Second Quarter

The effect pressure has upon cell reproduction and acid formation was determined at 1.9, 15, 20, and 30 psia at 30° and 40°C. The data showed that at 30°C pressures below and above 15 psia were detrimental to the formation of sulfuric acid and cell reproduction, while at 40°C no pressure was found under which the bacteria reproduced. It was demonstrated that at 40°C, pressures of 1.9 and 15 psia were bacteriostatic and pressures of 20 and 30 psia were bactericidal. To date, cultivation of T. thiooxidans for 5 weeks under a pressure of 30 psia at 30°C has not caused this organism to adjust to this pressure, contrary to earlier indications.

Temperature studies will be continued over the specified pressure ranges, with emphasis being directed toward any combinations of temperature and pressure that show promise.

### Status of Manuscripts

Biological Production of Sulfuric Acid, by Joseph A. Sutton, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Electrowinning of oxygen from silicate rocks  
Investigator: Thomas A. Henrie, Project Coordinator  
Location: Reno Metallurgy Research Center  
Reno, Nevada  
Date begun: June 1966 To be completed: May 1969  
Personnel: Thomas A. Henrie, Supervisory Research Metallurgist  
Donald G. Kesterke, Research Extractive Metallurgist  
Freddy B. Holloway, Physical Science Technician

## PROGRESS REPORT

### Objective

To determine the feasibility of obtaining elemental oxygen from silicate minerals by electrolytic methods, for use by the Earth inhabitants of the Moon. Emphasis will be directed toward the determination of essential physical and electrochemical properties of silicate and silicate-base melts containing various amounts of halide salts. Complementary investigations will be made to find suitable nonreactive crucible and anode materials for use in silicate melts, or in melts containing halides.

### Progress During the Second Quarter

Experiments to determine melting points and relative viscosities were initiated on a random mixture of rocks that included granite, rhyolite, and andesite. In the first test, 3,000 grams of this mixture were heated in a graphite crucible to 1,650°C under a helium atmosphere. A completely fluid melt was not achieved. In subsequent experiments, four additions of LiF, in 5-weight-percent increments, were made to serve as a flux. Addition of the first charge of LiF lowered the solidus temperature from about 1,165°C to 950°C and increased the fluidity of the melt. Each additional charge of LiF lowered the solidus temperature 40° to 45°C and further increased the fluidity. However, the melt was still too viscous to be used in an electrolytic cell at 1,300°C.

A second rock mixture that consisted of a basaltic material plus a pumice-like sinter was tested. Incipient melting of this mixture without added fluorides occurred near 1,000°C. At 1,200°C, the melt had a taffy-like consistency, and did not become relatively fluid until a temperature above 1,400°C was reached. Fluorite was then added in 5-weight-percent increments, to a total of 15 weight-percent. The greatest effect on the melt characteristics occurred with the first addition of  $\text{CaF}_2$ , which lowered the solidus temperature to about 900°C and gave the melt a more fluid consistency at 1,300°C. The presence of 10 weight-percent fluorite lowered the solidus temperature another 100 degrees and slightly increased the fluidity. No further increase in fluidity was achieved with 15 weight-percent  $\text{CaF}_2$ , and the overall result was that

the mixture was too viscous for use. Encouraging results were achieved in a duplicate series of experiments using LiF as the flux. The solidus temperature was decreased to about 675°C by the addition of 5 weight-percent LiF, but the melt was too viscous at 1,200° to 1,300°C. The solidus temperature of the mixture remained essentially the same with LiF contents of 10 and 15 weight-percent, but the bath fluidity increased markedly. Experiments are being conducted to determine whether these melts have sufficient current carrying capacity to allow their use as electrolytes.

During all experiments, periodic analyses of cell gases were made by infrared spectrophotometry. Major gases in the cell atmosphere were carbon oxides, particularly CO, originating from carbothermic reduction of rock constituents such as iron compounds. SiF<sub>4</sub> was formed in mixtures containing added fluorides at temperatures above 1,150°C.

#### Status of Manuscripts

Electrowinning of Oxygen from Silicates, by Donald G. Kesterke, was presented at the NASA review meeting at the Twin Cities November 30.

Task title: Stability of hydrous silicates and oxides in lunar environment  
Investigator: Hal J. Kelly, Project Coordinator  
Location: Albany Metallurgy Research Center  
Albany, Oregon  
Date begun: April 1966 To be completed: March 1968  
Personnel: Hal J. Kelly, Supervisory Ceramic Research Engineer  
Raymond L. Carpenter, Research Physicist

## PROGRESS REPORT

### Objective

The long-range objective is the determination of the energy requirements for dissociating silicate and oxide minerals to recover oxygen and/or water. The immediate objective is to investigate the stability under high vacuum and elevated temperature of some silicate and oxide minerals employing differential thermal analysis (DTA) and thermogravimetric analysis (TGA).

### Progress During the Second Quarter

The DTA furnace and thermocouples have been installed in the vacuum chamber. Test runs with the equipment indicated that extensive modification of equipment would be necessary in order to obtain thermographs that could be interpreted quantitatively. The original equipment used an alumina block, platinum crucibles, and shielded thermocouples. This arrangement did not have high enough sensitivity. Runs made with the crucible removed and with crucibles and unshielded thermocouples also gave low sensitivity. It appears that the use of crucibles in an alumina block will not give adequate sensitivity. Therefore, a setup similar to that described by S. L. Boersma will be used. This method has the advantage that no block is used, the samples are contained in metallic crucibles, the sensitivity is high and the heat of reaction is adsorbed mainly by the crucible, which makes calibration for heats of reaction possible. Preliminary tests with this method showed that adequate sensitivity could be obtained but base-line drift and noise became troublesome.

Modification of the furnace controls unit and the introduction of a grounded shield in the furnace has eliminated the noise problem. Base-line drift has not been completely eliminated, but rebuilding of the furnace has reduced it somewhat. Efforts will be made to reduce it more by the use of radiation shields.

Concentration and X-ray analyses of olivine minerals have been completed. Olivines with from 8 to 12 percent fayalite were obtained.

A sample of fayalite with a small amount of an unidentified compound has also been obtained. Three samples of micas, muscovite, Lepidolite, and biotite have also been obtained. These are the principal minerals to be used in this investigation.

#### Status of Manuscripts

Stability of Silicates and Oxides, by Hal J. Kelly, was presented at the NASA review meeting at the Twin Cities November 30.